Chapter 3

Experimental Technique and Data Analysis

3.1 Introduction

To obtain complete information about the dielectric behavior of the system, one needs measurements over a wide frequency range starting from few Hz to several GHz. Frequency domain techniques are usually used where wide range of frequency is required. But, this needs the highest degree of experimental expertise. Furthermore, information obtained is either at only one frequency or in a limited band of frequency. For example in X-band, information can be obtained in between 8.2 to 12.4 GHz only. Low frequency (MHz) range is not possible with this technique. For this purpose, one has to change the experimental setup at each time. In addition to these drawbacks, measurements using frequency domain techniques are time consuming, lengthy and large amount of material is required to fill in the cell.

Time domain spectroscopy (TDS) technique, on the other hand, to the study of dielectric relaxation process gives information in wide frequency range from few MHz to several GHz or even more in just a single measurement.

The enormous potential of this method is its simplicity of operation and gives relatively rapid estimation of the dielectric parameters usually associated
with a dielectric relaxation process. Measurements of dielectric relaxation studies with TDS are relatively faster. It has the advantage of requiring very small amount of material. This is important when measurements are to be made on expensive materials or materials that are available in small amount. Time domain can be considered as truly spectroscopic technique because of their broadband nature and their capacity to generate dielectric properties as continuous function of time or frequency.

TDS may be subdivided into two categories; time domain reflectometry (TDR) based on reflection method and time domain technique based on transmission method called as transmission dielectric time domain spectroscopy.

Use of TDR for the measurements of dielectric properties was first reported by Fellener-Feldegg [1] in 1969. Then Suggest A [2] were able to improve the original method leading to a higher accuracy. Fruitful attempts have been made to modify the TDS technique, to minimize errors in measurements and to increase the frequency range in the dielectric studies by Cole [3], Chanine and Bose [4], Gestblom and Noreland [5] described the transmission method in TDS. Cole [6] suggested a precision difference method for evaluation of dielectric behavior in time domain. Bevington et. al.[7] described a method, which allows estimates to be calculated for the unwanted reflection errors. Gestblom [8] suggested the single reflection method in TDS. Shannon [9] developed a lumped capacitor TDS technique. Cole et al [10] has developed an extremely powerful method of bilinear correction to eliminate the
reflections at higher frequencies. Patil and Mehrotra [11] have given sensitivity analysis of bilinear calibration method for TDR and proved that bilinear calibration method is effective than trilinear calibration method.

Recently, the research group [12,24], working at microwave laboratory, Department of Physics, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad has carried out research on number of chemically and biologically important binary liquid (solute + solvent) systems.

### 3.2 Basic Principle of TDR

In all time domain methods a fast rising step voltage is propagated in an ideal (low loss) coaxial line. The shape of step voltage remains unchanged as long as the propagation properties of the coaxial line remain same. When the line contains a section with different propagation properties, for example a section with dielectric material enclosed, the step voltage will partly be reflected and partly be transmitted at the interface.

The step voltage with fast rise time of the order of 20 pico-seconds as produced by pulse generator is detected at the sampling head of the sampling system and displayed on an oscilloscope. The step voltage is traveling to the dielectric sample filled in a cell, and reflected back which reaches the sampling head at some later time, is also displayed on the oscilloscope screen.

The current $I(\omega)$ and voltage $V(\omega)$ in a coaxial line with characteristics admittance $g_c$ ($g_c = 1/50$ for ideal 50 ohm transmission line) are related to incident and reflected voltage $v_0$ and $r_x$ by
\[ I(\omega) = g_c (v_0 - r_x) \]

and

\[ v(\omega) = (v_0 + r_x) \]

The input admittance \( y(\omega) \) of a termination is obtained from

\[ y_x = i/v = g_c (v_0 - r_x) / (v_0 + r_x) \quad (3.2) \]

In a particular case of the system exhibiting negligible conductivity, the admittance of the sample at the input of the transmission line is given by

\[ y_x = i\omega C g \varepsilon^*/Z \cot Z \quad (3.3) \]

where \( Z = (\omega d/c) \sqrt[2]{\varepsilon^*} \) and term \( Z \cot Z \) can be written to exploit the series expansion as

\[ Z \cot Z = 1 - (\omega d/c)^2(\varepsilon^*/3) - (\omega d/c)^4 (\varepsilon^*/45) + \ldots \ldots \quad (3.4) \]

where \( \varepsilon^* \) is the frequency dependent complex permittivity, \( \omega \) is the angular frequency, \( d \) is the length of coaxial line section filled with dielectrics and \( c \) is the speed of electromagnetic radiation in air and \( cg \) is geometrical capacitance with sample \([2\pi c'/\log(b/a)]\). From equation (3.2) and (3.3)

\[ \varepsilon^* = \left( g_c / i\omega C g \right) \left[ (v_0 - r_x) / (v_0 + r_x) \right] Z \cot Z \quad (3.5) \]

or

\[ \varepsilon^* = \left( c / i \omega d \right) \left[ (v_0 - r_x) / (v_0 + r_x) \right] Z \cot Z \quad (3.6) \]

These equation indicates that the dielectric permittivity of an unknown sample can be found if the time profiles of the incident and reflected pulses are known within a frequency range determined by the time limits of \( v_0 \) and \( r_x \).

Rearranging Equation (3.6) to get \( V_0 \), as
\[ V_0 = \frac{r_1[Z \cot Z + (i \omega d / c)]}{Z \cot Z - (i \omega d / c)} \]  

(3.7)

for air medium, \( \varepsilon^* = 1 \) and \( r_x = r_1 \) and equation (3.7) becomes

\[ V_0 = \frac{r_1[Z \cot Z + (i \omega d / c)]}{Z \cot Z - (i \omega d / c)} \]  

(3.8)

Equating equations (3.7) and (3.8)

\[ \frac{r_1}{r_x} = \frac{[Z \cot Z + (i \omega d / c)\varepsilon^*][Z \cot Z - (i \omega d / c)]}{[Z \cot Z - (i \omega d / c)\varepsilon^*][Z \cot Z + (i \omega d / c)]} \]  

(3.9)

Or

\[ \frac{C}{i \omega d} x \frac{r_1 - r_x}{r_1 + r_x} = \frac{\varepsilon Z \cot Z - Z \cot Z}{(Z \cot Z)^2 + (\omega^2 d^2 / c^2) \varepsilon^*} \]  

(3.10)

The quantities \( r_1 - r_x \) and \( r_1 + r_x \) will be called as \( p \) and \( q \) respectively.

Since Laplace transform is a linear operator,

\[ p(i \omega) = L \{ p(t) \} \]

and

\[ q(i \omega) = L \{ q(t) \} \]

Neglecting the quadruple resonance effect due to multiple reflections and using the approximation \( Z \cot Z = 1 \), we can write equation (3.10) as

\[ \frac{C}{i \omega d} x \frac{p(i \omega)}{q(i \omega)} = \frac{\varepsilon Z \cot Z - Z \cot Z}{(Z \cot Z)^2 + (\omega^2 d^2 / c^2) \varepsilon^*} \]  

(3.11)

The left hand side of this equation is called reflection function [5] which is a complex quantity given by
\[ \rho^*(\omega) = \frac{C}{i\omega d} \times \frac{p(i\omega)}{q(i\omega)} \]  

(3.12)

Therefore, equation (3.10) can be written as

\[ \varepsilon^* - 1 = \frac{ [1+ (\omega d / c)^2 ] \rho^* }{ [1- (\omega d / c)^2 \rho^* ] } \]  

(3.13)

Or

\[ \varepsilon^* - 1 = \frac{(1+A) \rho^*}{1-B \rho^*} \]  

(3.14)

Where \( A = B = (\omega d / c)^2 \)

From equation (3.14) one can easily determine complex permittivity \( \varepsilon^* = \varepsilon' - j\varepsilon'' \), provided that reflection function \( \rho^* \), \( A \) and \( B \) are known.

### 3.3 Reflections from various terminations:

In TDR technique a voltage step is propagated down the transmission line towards the sample under investigation and reflected voltage waves are monitored by oscilloscope at particular point on line. TDR measurements can give characteristics impedance of line and it shows both position and nature (resistive inductive or capacitive) of each discontinuity along the line. TDR also demonstrate weather losses in transmission line system are series losses or shunt losses. Furthermore, TDR measurement give meaningful information regarding broadband response of transmission line.
(a) **Propagation of signal along Transmission line**

The equivalent circuit for transmission line is shown in Fig. 3.1.

![Equivalent circuit of coaxial transmission line](image-url)

**Fig. 3.1 : Equivalent circuit of coaxial transmission line**

If C, G, L and R are defined per unit length for infinite long transmission line, then we can write

\[ Z_{in} = Z_o = \frac{\sqrt{R + j\omega L}}{G + j\omega C} \]  

(3.15)

Where \( Z_o \) is the characteristic impedance of transmission line.

A voltage pulse introduced at the input of transmission line requires finite time to travel distance ‘x’ along the line. The phase of the voltage moving along the line lags behind the voltage introduced at input by amount \( \beta \) per unit length. Furthermore, voltage will be attenuated by an amount \( \alpha \) per unit length by series resistance and shunt conductance of the line. The phase shift and attenuation are defined by propagation constant \( \gamma \) as

\[ \gamma = \alpha + j\beta = \frac{\sqrt{R + j\omega L}}{G + j\omega C} \]  

(3.16)

The velocity with which voltage propagates along the line can be written as

\[ V_p = \frac{\omega}{\beta} \text{ unit length per sec} \]  

(3.17)

The velocity of propagation approaches \( V_c \), for transmission line with air
dielectrics. For general case where \( \varepsilon_r \) is dielectric constant of medium.

\[
V_p = V_c / \sqrt{\varepsilon_r} \quad (3.18)
\]

The voltage and the current at any distance ‘x’ along the transmission line can be written in term of propagation constant \( \gamma \) as

\[
E = E_{in} e^{-\gamma x} \quad \text{and} \quad I = I_{in} e^{-\gamma x} \quad (3.19)
\]

Since the voltage and current at any point ‘x’ is known, characteristic impedance of the line can be written as.

\[
Z_o = \frac{E_x}{I_x} = \frac{E_{in} e^{-\gamma x}}{I_{in} e^{-\gamma x}} = Z_{in} \quad (3.20)
\]

When the transmission line terminated in a load whose impedance matches the characteristic impedance of the line, preceding equation satisfy the voltage and current relationship.

If \( Z_L \) is not equal to \( Z_o \) the incident energy is not fully delivered to load and propagates back towards source. The ratio of amplitude of reflected wave to incident wave is called reflection coefficient \( \rho \).

\[
\rho = \frac{E_r}{E_i} = \frac{(Z_L - Z_o)}{(Z_L + Z_o)} \quad (3.21)
\]

The end of transmission line will decide the value of \( Z_L \) and thus the value of \( \rho \). Thus \( \rho \) is related to the properties of material placed at the termination.
(b) **Step reflection from purely resistive loads**

The TDR technique uses reflected pulse from sample to determine dielectric parameters. The block diagram for step reflection from load is shown in figure 3.2

![Block Diagram](image)

**Fig. 3.2: The block diagram for step reflection from load.**

It is very interesting to observe response of different types of load to incident step. The shape of the reflected pulse is valuable since it reveals both, the nature and magnitude of mismatch. The typical examples for different values of purely resistive load are shown in figure 3.3. The knowledge of $E_r$ and $E_i$ measured on oscilloscope allows $Z_L$ to be calculated in terms of $Z_o$. 
Fig. 3.3: Reflection of step pulse from different purely resistive loads
Assuming \( Z_o \) to be real (as in precision cables), it is seen that, mismatch reflects a voltage of the same shape as the driving voltage, with magnitude and the polarity of \( E_r \) determined by relative values of \( Z_L \) and \( Z_o \).

Fig. 3.4 : Reflection of step pulse from different complex loads
(c) **Step reflection from complex loads**

The response of complex loads of different types to incident step pulse is shown in Fig 3.4. The reflected voltage from complex loads is evaluated at \( t=0 \) and \( t=\infty \), by assuming any transition between two points to be exponential.

### i) Series R-L

At \( t = 0 \) reflected voltage is \( + E_r(t) \). This is because inductor will not accept sudden changes in current and initially it look like infinite impedance. Then current in \( L \) builds up exponentially and its impedance drops down towards zero. At \( t = \infty \) \( E_r(t) \) is determined only by the value of \( R \).

\[
\rho = \frac{(R - Z_0)}{(R + Z_0)} \text{ at } t = \infty
\]  

The exponential transition of \( E_r \) has time constant determined by effective resistance seen by inductor. Since the output impedance is \( Z_0 \) in series with \( R \)

\[
\tau = \frac{L}{(R + Zo)}
\]  

The reflected voltage can be written as

\[
E_r = E_i \left[ \left( 1 + \frac{(R - Z_0)}{(R + Z_0)} \right) \left( 1 - \frac{(R - Z_0)}{(R + Z_0)} \right) e^{-t/\tau} \right]
\]  

### ii) Shunt R-C

At \( t = 0 \), load appears as short circuit since capacitor will not accept sudden changes in voltage and thus \( E_r = -E_r \). At \( t=\infty \), capacitor is effectively open circuit and \( Z_{\text{L}} = R \).

\[
\rho = \frac{(R - Z_0)}{(R + Z_0)} \text{ at } t = \infty
\]
The resistance seen by the capacitor is \( Z_0 \) in parallel with \( R \). Therefore the time constant of exponential transition is
\[
\tau = \left\{ \frac{Z_0 \cdot R}{(R + Z_0)} \right\} C \quad (3.27)
\]
The reflected voltage can be written as
\[
E_r = E_i \left\{ 1 + \frac{(R - Z_0)}{(R + Z_0)} \right\} (1 - e^{-t/\tau}) \quad (3.28)
\]

### iii) Shunt \( R - L \)

At \( t = 0 \), \( Z_L = R \) (assuming \( R > Z_0 \)) and at \( t = \infty \), \( Z_L = \infty \) impedance seen by inductor is parallel combination of \( R \) and \( Z_0 \). Thus,
\[
\tau = \left\{ \frac{(R + Z_0)}{Z_0 \cdot R} \right\} L \quad (3.29)
\]
The voltage \( E_r \) is given by equation.
\[
E_r = E_i \left\{ 1 + \frac{(R - Z_0)}{(R + Z_0)} \right\} (e^{-t/\tau}) \quad (3.30)
\]

### iv) Series \( R-C \)

At \( t = 0 \), \( Z_L = R \) (assuming \( R > Z_0 \)) and \( t = \infty \), \( Z_L = \infty \). Impedance seen by capacitor is series combination of \( R \) and \( Z_0 \). Thus
\[
\tau = (R + Z_0) \cdot C \quad (3.31)
\]
The voltage \( E_r \) is given by equation
\[
E_r = E_i \left\{ 2 \left[ 1 - \frac{(R - Z_0)}{(R + Z_0)} \right] \left( e^{-t/\tau} \right) \right\} \quad (3.32)
\]
3.4 Time Window and Source of Errors:

The choice of time window through which the reflected signals are observed has to be related according to the frequency range of interest. The lower limit of frequency spectrum contained in step pulse depends on time window used, while upper limit depend on rise time of pulse. The minimum frequency observable is \( F_{\text{min}} = 1 / (\text{time window}) \) while the maximum frequency observable is \( F_{\text{max}} = N / (2 \times \text{time window}) \) the number of points used to sample and digitize the signal.

For digitizing the signal it is necessary to select number of points per waveform in the time window. To reduce the noise an averaging of signal 16, 64 or 512 times can be done.

(a) Time referencing and truncation errors

When we observe the reflected pulses the reference point with respect to which we measure the amplitude of the pulse is an important factor. The time reference of reflected pulse from cell without sample should match with the time reference of pulse reflected from cell with sample. The improper time referencing may give errors in reflection coefficient. Generally, reflected pulses with sample and without sample do not have the same relative time position for various reasons. The effect of this error becomes serious at high frequencies. The correction of timing errors can be carried out in number of ways. One of the commonly used methods for proper time referencing is to extrapolate the linear portion of the pulse. The extrapolation method is simple but questionable
from the point of view of principles. Actually, in this method one tries to find out the fixed points from the base line, which is far from a straight line moreover the unevenness of the base line in this portion of the reflected pulse depends upon the reflecting medium.

Another major experimental difficulty in TDR measurement is the selection of start and finish points on the time domain waveforms for the Fourier transform. Because of the less than perfect pulse shape and the presence of unwanted reflections from impedance mismatches in the experimental system, truncation errors generates some errors in reflection coefficient over the whole frequency range.

(b) Unwanted reflections

In any real system extraneous signals arise from discontinuities and impedance mismatches in the coaxial line at the generator, sampling probe circuit etc. which are superposed on the designed reflection or transmission signals. Although the major sources of unwanted reflections are known in practice it is extremely difficult to determine their exact contribution in the reflection coefficient.

Most of the unwanted reflections arising from discontinuities and impedance mismatches in the measuring coaxial line must be kept outside the time window of interest by a proper choice of transmission line length. Further
using numerical smoothening techniques reduces the effect of unavoidable reflections in the portion in which they are present in the response signal.

(c) Drift

The various components of TDR equipment such as amplifier and scanning circuits have noise, which introduces errors in their output signals. Short time fluctuations in the time base circuitry also cause distortion of pulse. There is also a drift of the pulse with respect to time. The use of proper warm up time reduces errors in measurement due to drift in pulse.

3.5 The Developed Setup of TDR Unit:

Actual photograph of the developed system is shown in fig 3.5. The experimental setup shown in fig.3.6 consists of sampling oscilloscope DS1000[25], with TDR module, a transmission line, and sample cell. All these components in experimental setup are discussed in following sections.
Fig. 3.5: The Photograph of developed TDR unit

Fig. 3.6: Photograph of experimental setup with developed TDR unit
The schematic layout of various components in the experimental setup with developed TDR unit is shown in Fig 3.6b.

Fig. 3.6b: Block diagram of Dual channel TDR unit

1. Mainframe TDR unit
2. DS 1000 oscilloscope
3. Pulse generator 3A. Loop through conn.
4. Sampler holder /Sample Cell
5. Transmission lines (56 ohm)
6. Computer
A) DS1000 Oscilloscope.

The simplified block diagram of the developed TDR unit is as shown in the fig. (3.6). DS1000 sampling oscilloscope[31] is very precise instrument for digital data acquisition of instantaneous signals. The working of instrument depends upon front panel keys as well as menus of function are displayed along the right side of display screen. These menus are called soft key menus. Soft key menus list functions other than those accessed directly by the front panel keys. Pressing unlabeled key immediately next can access a function on soft key menu.

The front panel has knobs and buttons. The knobs are used most often and are similar to the knobs on other oscilloscopes. The buttons not only use some of the functions directly but also bring up soft button means on the screen, which gives access to many measurement features associated with advanced functionalities, math, and reference or run control features.

**Annotation on the screen:** The unlabeled keys next to the annotation on display are called soft keys. Front panel of the instrument includes a display area and several functional areas, which includes control, storage, Auto scale, Enter, Devices, Setup and System. The control section includes three keys Clear display, Run and Stop signal. These keys are used to clear screen, start data acquisition and stop data acquisition respectively.

The main features of DS1000 Digital Storage oscilloscope are

- Mono/Color TFT LCD Displays at 320*234 resolution
• USB storage and printing supports, firmware upgradeable via USB connectivity

• Adjustable waveform intensity, more effective waveform viewing

• One-touch automatic setup for ease of use (AUTO)

• Saves 10 Waveforms, 10 setups, supports CSV and bitmap format

• Delayed Scan Function, easy to give attention to both details and overview of a waveform

• 20 Automatic measurements

• Automatic cursor tracking measurements

• Waveform recorder, record and replay dynamic waveforms

• User selectable fast offset calibration

• Built-in FFT function, Frequency Counter

• Digital filters, includes LPF, HPF, BPF, BRF

• Pass/Fail Function, optically isolated Pass/Fail output

• Add, Subtract and Multiply Mathematic Functions

• Advanced trigger types include: Edge, Video, Pulse width, Slope, Alternative, Pattern and Duration (Mixed signal oscilloscope)

• Adjustable trigger sensitivity.

DS1000 series oscilloscopes provide an easy-to-use user interface, the definitions of the buttons and the knobs are as follows:

**Menu buttons:** Associated with Measure, Cursor, Acquire, Display, Storage, and Utility menus.

**Vertical buttons:** Associated with CH1, CH2, MATH, REF and LA menus, the OFF button can set waveform or menu which currently active off.

**Horizontal buttons:** Associated with horizontal MENU.
**Trigger buttons:** Associated with trigger MENU, instant action to set 50% trigger level and FORCE trigger.

**Action buttons:** Include run control buttons for AUTO and RUN/STOP.

**Function buttons:** Five grey buttons from top to bottom on the right to the LCD screen, which set choices of operation in the currently active menu.

Waveform key is used to store current waveform in memory of oscilloscope. Setup key is used for setting waveform. Print key is used to print current waveform or waveform in memory. Auto scale section contains only single key ‘auto scale’. This ‘auto scale’ key causes the instrument to quickly analyze the signal. Then, it sets up verticals horizontal and trigger to best display that signal. Entry devices are the keypad, the arrow keys and the knob. Entry devices can change the numeric settings of some soft keys, such as trigger level or to select an item from the list of choices. The set up section includes seven keys, Time base, Trigger Acquisition, Display, Marker, Define means and Math. With time base key we can change horizontal position of waveform and also the time window. Trigger can be used to change trigger level of signal. Acquisition key is used to set number of data acquisition points and also number of times the averaging is done. Marker key can be used for setting markers on waveform during measurement of specific parameters. One can also put markers (measurement marker lines) during measurement. Math function is used to perform few mathematical operations such as addition and subtraction of two waveforms or even Fourier transform of waveform. Additional functions listed in bold type above and below some of the front
panel keys. These functions are called shifted functions. Pressing front panel shift key and front panel key next to the desired function can activate these shifted functions.

**The DS 1000 has the following hardware:**

The major hardware components of DS1000 oscilloscope are described below:

(a) **Interface circuitry**

The interface circuitry accepts commands from system microprocessor to control the module functions such as IF gain, sampler bandwidth and optical channel bandwidth.

(b) **A/D Converter**

The A/D converter traces the peak value of the analogue pulse at it’s input and converts it into a 8 bit digital word. These 8 bit words are put into a FIFO memory, which is then read and processed by the CPU.

(c) **Fast-Cal:**

When turn on the fast-cal function, oscilloscope will calibrate vertical offset several times according to the running time. The Fast-Cal will run immediately when it’s turned on.

(d) **Self-Cal:**

Oscilloscope will calibrate parameter of vertical system (CH1, CH2, and Ext), horizontal system and trigger system.

(e) **Host RAM and Flash ROM**

The host RAM is 1 MB non-volatile RAM. This is where the waveforms data is held and manipulated. It also controls front panel set up, set up
memories and waveforms memories. The flash ROM contains the system firmware that controls operation of the instrument.

(f) User Interface

DS1000 series oscilloscopes provide an easy-to-use user interface, the definitions of the buttons and the knobs.

(g) Ports

The centronics port is a parallel connector for printers compatible with centronics interface.

(h) Video RAM and Display

The system uses 1MB fast video RAM for storing the display image. The video RAM also contains the pixel memory. The display is 9 inch, high resolution, and color display.

(i) USB Interface

Supports standard USB interface and can be used with external non volatile and flash memory devices for file operations.

B) Pulse generator

The pulse generator circuit has been developed by using Schmitt trigger circuit. The frequency of generated pulses is 4.5 KHz with pulse width of 6 μs. The rise time of the pulse is 5 ns and amplitude is 42 mV. Along with the pulse signal, the trigger signal is also generated. This trigger signal is used as external trigger to DS1000 oscilloscope.
C) Sample holder

The sample holder is the important and critical part of the setup. The sample holder designing depends on type of sample under study. The commonly used sample holder with coaxial cable is SMA type. As the SMA connectors have already designed for precise 50 Ω impedance. So that, the coaxial line is such that its characteristic impedance is

\[ Z = \frac{138.2}{\sqrt{\varepsilon}} \log_{10} \left( \frac{b}{a} \right) \]

This impedance of our transmission line is 56Ω. Here ‘a’ is inner diameter of outer conductor and ‘b’ is diameter of inner conductor and ‘\( \varepsilon \)’ is relative permittivity of the dielectrics between the conductors.

![Sample holder](image)

**Fig.3.7 : Sample holder**

The sample cell holds the liquid. The physical dimensions of the cell are very important. So one must be careful while designing the sample cell. The impedance of the cell should be matched with coaxial transmission line to which cell is connected. If there is impedance mismatch then unwanted reflections may disturb the wave thereby causing some errors in measurement. The proper design of cell includes the inner conductor and outer conductor diameters. The length of inner conductor is called as pin length of the cell and
is very important factor in analysis. The sample length must be enough to avoid unwanted reflections.

In total reflection method the sample length must be long enough to produce an adequate difference signal but short enough to keep less complication of resonance effects at frequencies above the range of interest.

3.6 TDR Waveforms:

The complete experimental setup of TDR system is shown earlier in the Fig3.6. The instrumentation involved is explained in an earlier section A flexible coaxial cable of about 0.45m length is connected between TDR unit and sample holder. The measuring sample cell used is a indigenous cell designed and constructed in the laboratory as shown above in Fig.3.7.

The incident and reflected waveforms are as shown in fig.3.8

![Incident and Reflected Pulses](image)

Fig.3.8: Photograph of incident and reflected pulses
Considering the importance of time window, to reduce unwanted reflections it is kept to 60 ns. It can be changed depending on the nature of dielectric liquid under study. For the digitization of pulse, 600 points per waveform are used. Only reflected pulse is viewed in the time window of 60 ns on the screen.

Fig. 3.9a : Reflected pulse from empty cell

Fig. 3.9b : Reflected pulse from cell with tap water
The reflected pulse $R_1(t)$ from the sample cell without sample is as shown in fig. 3.9a and the reflected pulse $R_s(t)$ from the sample with tap water is shown in fig 3.9b.

**Fig. 3.10 : Reflected waveforms recorded without sample $R_1(t)$ and with the tap water $R_x(t)$**

### 3.7 Experimental procedure and Data acquisition:

The TDR unit is used for measurement after warming up for at least 30 minutes. This is necessary to get setup pulse without drift. A fast rising voltage pulse of 42mV with 5 ns rise time was propagated through a coaxial line. The sample was placed in the cell connected at the end in a coaxial transmission line. The reflected waveform is observed carefully. The unwanted reflections in the reflected pulse at point of contact between transmission line and TDR unit, as well as transmission line and sample cell is minimized by ensuring proper
contact between these components. The pulse reflected from the sample was monitored. A photograph of the experimental setup is shown in Fig 3.11.

Fig. 3.11 : Experimental setup to record reflected pulses
A time window of 60 ns was used. The reflected pulse from the channel without the sample R1 (t) and with sample Rx (t) were digitized with 600 points and saved on the hard disk of the computer attached to the experimental setup. A graph of typical reflected waveforms recorded without sample and with sample are shown in Fig 3.12a and fig.3.12b.

Fig. 3.12:(a) Reflected pulse from empty cell b) Reflected pulse from filled cell

**Fourier transforms:**

The data received by PC from TDR unit is in the form of voltage with respect to time. The combined file of R1(t) & Rx(t) is called as wfm file. The combined waveforms are shown in fig.3.13.

Fig. 3.13 : Waveforms of wfm file.
Addition and subtraction of pulses $R_1(t)$ and $R_x(t)$ is done to get $p(t) = R_1(t) - R_x(t)$ and $q(t) = R_1(t) + R_x(t)$. The baseline of both the waveforms is scaled to 0. The file containing $p(t)$ and $q(t)$ is called ‘tdr’ files. Thus tdr file contains the array of data points of $p(t)$ and $q(t)$. The tdr file waveform is as shown in fig.3.14.

![Waveforms of tdr file](image)

**Fig. 3.14 : Waveforms of tdr file.**

This time domain data is converted to frequency domain data using Fourier transform. While performing Fourier transform one should be careful about the nature of the curve for which transform is to be obtained. Since nature of curves $p(t)$ and $q(t)$ is different, separate methods of Fourier transform are used.

The Fourier transform of $p(t)$ is obtained by a summation method[27,28] using equation
\[
N \sum_{n=0}^{N} \exp(-j\omega nT) p(nT) = T
\]

The Fourier transformation using summation method has some limitations that for all the sampling intervals \( T \) the nature of pulse form must be known. Furthermore, the transform \( p(\omega) \) is simply the area under the curve \( p(t) \) which has an initial peak followed by a decay to zero or a finite limiting value strictly to infinite time.

The pulse form of \( q(t) \) is not known exactly. The \( q(t) \) rises monotonically to a long time limit. Therefore summation method of Fourier transform can not be used for \( q(t) \) curve. The Fourier transform of such type of curves can be done with the Samulon method \([29]\) for which expression is as follows;

\[
q(\omega) = \left[ \frac{T}{1 - \exp(-j\omega T)} \right] \sum_{n=0}^{N} \left[ q(nT) - q(n-1)T \right] \exp(-j\omega nT)
\]

In equation (3.33) and (3.34), \( \omega \) is angular frequency, \( T \) is the sampling interval or time difference between two adjacent points and \( N \) is number of points. For example if time window is 60ns then \( T \) is 100 ps for 600 points per waveform. The frequency domain data obtained from Fourier transform is used further to calculate complex reflection function \( \rho^*(\omega) \). The complex reflection function is given by equation (3.12).

\[
\rho^*(\omega) = (c/\omega d) \times \frac{p(\omega)}{q(\omega)}
\]
Where \( c \) is the velocity of light, \( \omega \) is angular frequency, \( d \) is effective pin length and \( p(\omega) \) and \( q(\omega) \) are Fourier transforms of subtracted and added pulses respectively.

The frequency dependent spectra is as shown in fig.3.15. The file containing this data is called as ‘fdr’ file.

![Complex reflection spectra for methanol at room temp.](image)

**Fig. 3.15 : Complex reflection spectra for methanol at room temp.**

The demands on the accuracy in \( \rho^*(\omega) \) are quite severe at high permittivity and high frequencies and the method has been mostly used for liquids of medium permittivity. The complex reflection coefficient spectra is called as ‘raw’ data. Using this data complex permittivity can be determined as follows.
The basic equation for determining relative complex permittivity $\varepsilon^*(\omega)$ of the sample derived from transmission line theory is conveniently written in simple form as

$$
\varepsilon^*(\omega) - 1 = \frac{C}{\iota \omega d} x \frac{(r_0 + r_x)}{(r_0 + r_x)} Z \cot Z
$$

(3.35)

Where $Z = (\omega d / C) \sqrt[\varepsilon^*]$ and $r_o$ and $r_x$ are the reflected pulses from the cell without sample and with sample respectively.

If we consider only single reflection, then $Z \cot Z = 1$ and equation (3.35) can be written as

$$
\varepsilon^*(\omega) - 1 = \frac{C}{\iota \omega d} x \frac{(r_o - r_x)}{(r_0 + r_x)}
$$

(3.36)

Equation (3.36) indicates that the dielectric constant of unknown sample can be found if the time profile of the incident $r_0$ and reflected $r_x$ pulses are recorded within a frequency range. A working equation more convenient than equation (3.36) can be obtained as follows by rearranging it.

$$
\frac{r_o}{r_x} = \frac{\varepsilon^* + (c / \iota \omega d)}{(c / \iota \omega d) - \varepsilon^*}
$$

(3.36a)

If the sample cell is without any liquid i.e. with air is considered, then

$\varepsilon^* = 1$ and we get

$$
\frac{r_o}{r_x} = \frac{1 + (c / \iota \omega d)}{(c / \iota \omega d) - 1}
$$

(3.37)
Where \( r_1 \) is reflected pulse in case of air with \( \varepsilon = 1 \). Eliminating \( r_0 \) from equations (3.36a) and (3.37).

\[
\frac{r_1}{r_x} = \frac{\varepsilon^* + (c / i\omega d)}{(c / i\omega d) - \varepsilon^*} \times \frac{(c / i\omega d) - 1}{1 + (c / i\omega d)} \tag{3.38}
\]

The equation can be rearranged and substituting \( \frac{C}{i\omega d} \times \frac{r_1 - r_x}{r_1 + r_x} = \rho^* \) to get,

\[
\varepsilon^* - 1 = \frac{\{1+ (\omega d / c)^2\} \rho^*}{\{1- (\omega d / c)^2 \rho^*\}} \tag{3.39}
\]

\[
\varepsilon^* - 1 = \frac{\{1+ A\} \rho^*}{1+ B\rho^*} \tag{3.40}
\]

Where \( A = B = (\omega d/c)^2 \)

Thus using equation (3.40) one can obtain complex permittivity spectrum in the desired frequency range. Using this complex permittivity spectrum static dielectric constant \( (\varepsilon_0) \), dielectric constant at infinite frequency \( (\varepsilon_\infty) \) and relaxation time \( (\tau) \) can be calculated by using Havriliak Nigami \[30\] expression.

\[
\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_0 - \varepsilon_\infty}{(j\omega\tau)^{1-a}} \tag{3.41}
\]
In the developed TDR unit due to limitation of generated pulse rise time of 5 ns, relaxation time could not be calculated for fast relaxing molecules of the liquid. The setup can be used for calculation of relaxation times of slow relaxing molecular liquids such as polymers and biological systems. Whereas, the permittivity and static dielectric constant can be calculated accurately. We have tested many polar liquids for calculation of dielectric constant and found the accurate values within 5% error compared to standard values.

Following table shows the values measured using this setup and standard values of some polar liquids.

<table>
<thead>
<tr>
<th>S.N</th>
<th>Liquid name</th>
<th>Static Dielectric Constant at room temp</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Standard</td>
</tr>
<tr>
<td>1</td>
<td>Water</td>
<td>80</td>
</tr>
<tr>
<td>2</td>
<td>Methanol</td>
<td>32.6</td>
</tr>
<tr>
<td>3</td>
<td>Propanol</td>
<td>20</td>
</tr>
<tr>
<td>4</td>
<td>PEG200</td>
<td>17.9</td>
</tr>
<tr>
<td>5</td>
<td>Acetonitrile</td>
<td>37.5</td>
</tr>
<tr>
<td>6</td>
<td>Ethanediol</td>
<td>37.7</td>
</tr>
</tbody>
</table>

3.8 TDR setup for Blood samples

Our aim is to develop TDR setup for pathological tests purpose. Generally, the pathological test samples are in small quantity. The SMA type cell need more sample. Also for viscous sample, it is difficult to clean the SMA type cell. It is necessary to develop a cell, which requires less sample and easy to clean.
Instead of using SMA type of design, we have used extended coaxial cable terminated in strip with small gap less than 1 millimeter. This cell requires one or two drops of liquid sample and easy to clean. This cell can also be used for gels, more viscous liquids and biological samples.

The first setup prepared is for blood samples. In the present case, the liquid under test is blood and is a thick viscous liquid that clots on standing, therefore, keeping in mind other constraints discussed earlier the cell was designed in flat rectangular planar geometry using standard copper clad sheet. Schematic lay out of the geometrical construction of the sample cell is shown in Fig.3.16. It is constructed using standard copper clad sheets used in printed circuit boards.

![Copper Plate](image)

**Fig. 3.16 Geometrical construction layout of sample cell made from copper clad sheet.**
References:


